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Characteristics of a pulsed packed, liquid-liquid extraction column.

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CHARACTERISTICS OF A PULSED, PACKED, LIQUID-LIQUID EXTRACTION COLUMN

Leonard Pinzow

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LIQUID-LIQUID EXTRACTION COLUMN

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CHARACTERISTICS OF A PULSED, PACKED,

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MASTER OF SCIENCE

IN

MECHANICAL ENGINEERING

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ABSTRACT

An investigation was conducted of the effect of varying water flow rates and pulse frequency on the performance of a pulsed, packed, liquid extraction column, using the system benzoic acid, toluene, and water. Water was maintained as the continuous phase. It was found that in the range of parameters considered, that the higher the frequency of pulsation and the higher the water flow rate, the lower the height of a transfer unit. The heights of the transfer unit calculated for the conditions considered were 0.97-2.73 feet when referred to the water side and 8.83-40.5 feet when referred to the toluene side for pulse frequency amplitude products of 13 to 65 inches/minute. This represents a considerable improvement over values listed in the literature for unpulsed columns.

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1. Introduction.

  Liquid-liquid extraction is a process in which a substance, dissolved in one liquid phase, is transferred to another liquid phase, which is partially or totally immiscible in the first liquid. The substance dissolved in the first phase is called the solute and the solution of solute in the first phase is called the feed. The liquid to which the solute is transferred is called the solvent. The extraction procedure consists of effecting an intimate mixture of the solvent and feed during which time the transfer of solute takes place. The solute rich solvent, which may be referred to as the extract, must then be separated from the depleted feed. The latter is called the raffinate. The process is then completed by the recovery of the solvent from the extract and the raffinate. This may be accomplished by one of the unit operations such as distillation, or by chilling or heating to reduce the solubility of the solvent or solute.

  The objective of liquid extraction is separation. Either it is desired to remove a small amount of unwanted material or to obtain some product from the feed. The selectivity of the solvent, chosen to achieve this aim, can be expressed in terms of the separation factor, which is the ratio of the distribution coefficients of the desired and undesired materials when equilibrium is attained between the two phases. The distribution coefficient is the ratio of the concentration of a component in one phase to its concentration in the other phase at thermodynamic equilibrium. The distribution coefficient varies both with temperature and concentration. The curve showing the variation of concentration of the two phases for a particular temperature is called the equilibrium curve. This curve is a measure of the maximum amount of material which can be extracted and therefore can be used to guage the effectiveness of a particular extraction procedure. For low concentrations, the equilibrium curve is practically

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a straight line and the variation with temperature is small.

2. Fields of Application.

The fact that the desired product is still in solution at the end
of the extraction, and requires separation from the solvent, means that in
order to economically justify its utilization, some impelling reasons must
exist. It is now being applied to large tonnages in the petroleum, phar-
maceutical, and nuclear power fields. Some of the reasons for its use are:

a. The relative volatilities of the components of a mixture are close
to unity. In this case separation by distillation would involve the use
of a column with an excessive number of plates. Extraction into a system
with a greater difference of volatilities would permit the use of a smaller
column with a resulting decrease of overall cost. The Edeleanu process
has been developed for this reason and is used in the refining of light
lubricating oils and diesel oil. It utilized liquid sulfur dioxide to
remove aromatics from paraffinic and naphthenic hydrocarbons with similar
boiling points in petroleum.

b. When application of elevated temperatures would damage the desired
product, it is sometimes possible to extract into a system which permits
ultimate separation at lower temperature. Vitamins, as well as unsaturated
oils, both of which are temperature sensitive, are extracted from such fish
as cod and menhadden. In the pharmaceutical industry such antibiotics as
penicilin and bacitracin, which are also temperature sensitive are extract-
ed from fermentation liquor. The former is extracted by amyl acetate, the
latter by butanol.

c. When a small amount of non-volatile materials is dissolved in a
large quantity of solvent, it may be feasible to use extraction to provide
a much more concentrated solution which could be separated more economically.
Liquid propane is used to dewax and deasphalt crude oils. The liquid pro-

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pane is refrigerated and kept under pressure during the extraction, and is vaporized from the extract, permitting it to be recovered for recycling.

d. Separation of two or more non-volatile solids cannot be achieved by distillation. They may be dissolved and separated by solvent extraction. The solvent selected is one in which the distribution ratio for the material to be extracted is greater than that for the material remaining behind in the raffinate. An example is the elimination of sodium chloride from a 50% solution of sodium hydroxide by the use of liquid ammonia.

In the nuclear power field, liquid extraction has been applied extensively. This is true because the process lends itself to operation by remote control and because very high purity is achieved in the product. Liquid extraction is used in the concentration of uranium ore as well as in the separation of plutonium and uranium from fission products in spent fuel. The separation depends on the fact that uranium and plutonium, in a high oxidation state, are more soluble in organic solvents than the fission products, when a salting agent is present. Thus a fuel element may be dissolved in nitric acid and the uranium and plutonium extracted with an organic solvent. The plutonium can then be removed by reducing it to a lower oxidation stage, which reduces its solubility in the organic solvent and permits it to be extracted with water. Finally, in the absence of the salting agent, the uranium can be stripped with water from the organic solvent which is then recycled.

Extraction cannot be used if the feed and the solvent have too high a degree of mutual solubility or if they have the tendency to form a stable emulsion. Since most of the separations are accomplished by gravity settling, it is desirable to have as high a difference in the density of feed and solvent as possible. In addition, if the solubility of the solute in the solvent is very low, a great deal of solvent may be

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required for the extraction. This may make the process extremely costly from the standpoint of high energy requirements for the pumping and recovery of the solvent.

3. Methods of Operation and Equipment.

Basically, the method of achieving the intimate mixture of the solvent and feed necessary for bringing about mass transfer, is by providing some turbulent action by means of which one phase is broken up into droplets and passed through the other, which remains the continuous phase. The amount of material transferred from one phase to the other is proportional to the interfacial area as well as the concentration gradient which exists across the film boundary. Turbulence, by promoting the formation of many small droplets, promotes a large interfacial area and in addition increases the amount of material transferred from the film to the body of the liquid by eddy diffusion. The importance of the formation and reformation of the droplets is emphasized by Sherwood's estimate that about 40% of the material extracted from a single drop passing through a column of liquid is removed during its formation.[1] An important factor which limits the amount of turbulence which can be applied to a system is the necessity of separating the extract from the raffinate. If the interfacial tension is too low, a stable emulsion may be formed; if it is too high, there will be difficulty in dispersing one phase within the other. Other factors which affect the ease of mixing and separation are the densities, and viscosities of both phases.

The methods and equipment which have been used for liquid extraction can be broadly classified as follows:

    a. Batch method

    b. Mixer-settler system

    c. Continuous counterflow column

d. Mechanically assisted extractors

Many variations of each of these basic types have been patented.
The batch system is the simplest method, but the amount which can be ex-
tracted in one operation is limited. To increase the amount of material
extracted, as well as to permit more automatic operation, a continuous
counterflow system such as the mixer-settler may be used. The continuous
counterflow column is a unit containing spray equipment, packing, perforat-
ed plates or baffles which assist the extraction by increasing the inter-
facial area between the phases, either by droplet formation or by extending
the flow path. The mechanically assisted extractors provide additional mix-
ing energy which causes turbulence, thereby assisting the extraction by the
formation and reformation of droplets.

The batch process involves mixing a fixed portion of solvent and feed
and permitting them to settle into extract and raffinate layers which can
then be separated. The mixing is accomplished by mechanical agitation.
Mixing time is short; on the order of one minute. Separation is by gravity,
or in some cases where there is a small density difference or a tendency
to emulsify, centrifuges may be used. The settling time ranges from one-
half hour to one hour and depends on the design of the settler. The
effectiveness of extraction is high and may be judged on the basis of
whether or not equilibrium exists between the two phases leaving the tank.
This can be determined from the equilibrium curve.

The mixer-settler system is an example of the multiple stage continu-
ous type of operation. Each stage consist of a mixer and a settler. The
mixer may contain baffles and is fitted with some mixing device such as
a turbine, pump or nozzle. The settler is designed to minimize turbulence
of the entering and exiting streams. The mixer-settler stages may be
combined in numerous different arrangements with regard to the flow between

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stages, but the counterflow arrangement is the most common. The continuous counterflow mixer-settler has advantages over the batch method in that it lends itself more easily to automatic or remote control, but it has the disadvantage of large initial investment and floor space requirements.[2]

The spray column is the simplest type of continuous counterflow column. It consists of a cylindrical vessel with nozzles or a distributor head for introducing the dispersed phase. The effectiveness of the extraction depends on the prevention of coalescence of the dispersed phase and recirculation of the continuous phase. The advantages are low cost, high capacity and ease of cleaning.

The packed column provides a much larger area to promote contacting of the two phases, as well as preventing recirculation of the continuous phase, which is a problem in the spray column. Either liquid may be maintained as the continuous phase, but it is desirable to make the one with the higher flow rate continuous.[3] The interface between phases is maintained in either the top or bottom settling section of the column.

The packing consist of small pieces which are made in many different shapes and of a variety of materials. Commonly used shapes are Raschig Rings and Berl Saddles. The materials may be stone, ceramic, stainless steel, wood, coke, or carburundum. The packing material is selected on the basis of its ability to be preferentially wetted by the continuous phase. Otherwise, the dispersed phase will rise in rivulets rather than droplets and the column effectiveness is materially reduced. Other factors involved in the selection of the material are cost, strength, and chemical resistance to the feed and solvent.

The packed column has the advantage of simple construction and low cost. It has the disadvantage of tending to trap solids, clogging, and being difficult to clean.

The perforated or **sieve** plate type of column is one which uses the principle of redistributing the dispersed phase into droplets a number of times by passing it through a series of plates with a large number of small holes. In effect this corresponds to placing a number of short spray columns in series and taking advantage of the fact that a large portion of the extraction takes place during the formation of the drops. In operation, the dispersed phase collects under or above each plate until sufficient pressure is built up to force the liquid through the holes and form droplets which pass to the next plate. The continuous phase passes from plate to plate by means of downcommers. Decreasing the plate spacing increases the effectiveness of extraction but decreases the allowable throughput. The effectiveness of the sieve type column is high and it has the advantage of being fairly simple to construct. They can be built with diameters as great as twelve feet and thus have a large capacity. The disadvantages are that the perforations have a tendency to clog and that the column cannot be operated over as wide a range of flow rates as the other types of columns.

In all of the previously described countercurrent extractors, the mixing energy was provided by the difference in density of the continuous and dispersed phases. In many cases, this difference is small. In order to increase the effectiveness of extraction, mechanically supplied energy has been utilized. An example of this is the pulsed column which is based on the patent issued to Van Dijck[4] in 1935. His method consisted of moving a number of perforated plates up and down in an unpacked column, thereby providing relative motion between the column and its liquid contents and the plates. Alternatively, the column could be constructed as a sieve plate column and the liquid contents pulsed up and down with respect to the plates. The latter method has found a great deal of application in recent years, particularly in the processing of uranium and plutonium.

Several different methods of pulsing the liquid contents of the column have been devised. Cohen and Beyer[13] used a diaphragm pump pulsator as well as a cam driven bellows. Both of these were connected to the column by tubing. Feick and Anderson[5] utilized a neoprene diaphragm at the bottom of the column actuated by a rod going to an eccentric. The latter column utilized packing rather than sieve plates and improvement of from 4 to 10 times the effectiveness of the extraction of an unpulsed column was reported. Chantry, VonBerg, and Wiegandt[6] reported that the height of packed selection required to achieve an equivalent extraction is reduced by as much as three times when pulsing is applied to a packed column.

4. Theoretical Development.

If a concentration gradient exists within a single phase, there will be molecular diffusion from the point of high concentration to the point of low concentration. The amount transferred and the rate will depend on the diffusion coefficient of the liquid, the driving force, and the area perpendicular to the path of diffusion. Fick's law may be used to relate these quantities as follows:

$$\frac{N}{\theta} = Da'\frac{dC}{dL} \tag{1}$$

If turbulence is established within the fluid, there will be a great increase in both the amount and rate of transfer. The mechanism in this case is called eddy diffusion. The coefficient of eddy diffusion, E, can be added to D and for dilute solutions equation one can be integrated to yield

$$\frac{N}{\theta} = \frac{(D+E)a'(C_1-C_o)}{L} \tag{2}$$

The expression $\frac{(D+E)a'}{L}$ can be replaced by a single expression k, which is the mass transfer coefficient, and (2) becomes

$$N/\theta = ka'(C_1-C_o) \tag{3}$$

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When two immiscible fluids are in contact, there will exist concentration gradients in both phases which act as driving forces for the transfer of material from one phase to the other. The two film theory has been proposed by Whitman[7] to explain the mechanism in this case. It postulates the existance of a laminar film on either side of the interface between the two phases. Transfer within the bulk of each phase is primarily by means of eddy diffusion. Transfer across each film is by means of molecular diffusion with equilibrium existing at the interface. For steady state conditions, the concentration gradient established is as shown in Figure 1. The symbols used, refer to toluene and water which was the system investigated in this thesis, but can be applied to any two immiscible phases t, and w.
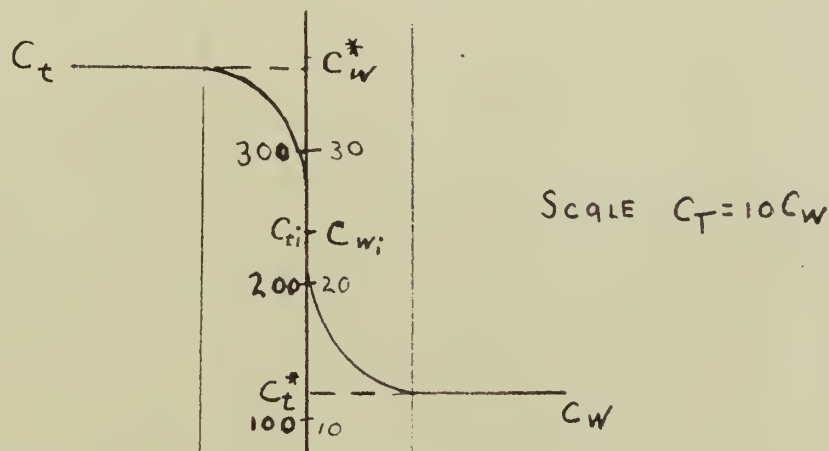
Figure 1. Concentration gradient across interface.

In the t phase the concentration of the main body of the fluid is C_t and the driving force across the thin film is $C_t - C_{ti}$. The concentration at the interface C_{ti} is in equilibrium with the concentration C_{wi}. The relationship between them may be found from the equilibrium curve. The driving force across the w phase film may be taken as $C_{wi} - C_w$. The actual value of C_i cannot be determined always and it is necessary to find some means of evaluating the driving force in order to find a solution to equation (3). In many cases, the magnitude of one or the other of the driving forces $C_i - C_w$ is much lower than the other. This means that the resistance

of the other film is controlling and that the rate equation may be evaluated
with respect to the controlling film side as follows. If phase t is the
controlling phase, $C_{wi} \cong C_w$. Since

$$C_{ti} = MC_{wi} \qquad\qquad (4)$$

it follows that $\qquad\qquad C_{ti} \cong MC_w$

The value, which is actually used in the rate equation, is C_t^* which is
defined by $\qquad\qquad C_t^* = MC_w \qquad\qquad (5)$

If phase w controls, $\qquad\qquad C_t = MC_w^* \qquad\qquad (6)$

For a continuous countercurrent column, these values may be substituted
into the rate equation, if the simple distribution law holds, i.e. if the
equilibrium curve is a straight line.

Since the total interfacial area a' is unknown, it is replaced by aV,
where a is the interfacial area per unit volume of the column and V is the
volume of the column. The a is then absorbed into the k as an overall mass
transfer coefficient and is written K_wa or K_ta, depending upon which phase
it is referred to. Since the concentration gradient varies continually
with the height of the column, and dv = sdZ, the rate equation (3) can now
be written in differential form as follows:

$$\frac{dN}{\theta} = K_wa \; (C_w^* - C_w)SdZ = K_ta \; (C_t - C_t^*)SdZ \qquad\qquad (7)$$

As shown below, this equation can be evaluated by integrating and substitut-
ing a logarithmic mean driving force, if the equilibrium curve is a straight
line, and the mass flow rates of the two phases remain constant throughout
the column. The latter can assumed to be true in the case of dilute
solutions, and when both phases are mutually saturated with each other.
If W, and T represent the flow rates of the two phases expressed in cubic
feet per hour, the material balance may be represented by

$$W(C_w - C_{w_1}) = T(C_t - C_{t_1}) \qquad\qquad (8)$$

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or
$$C_t = \frac{W}{T}C_w - (WC_{w_1}/T - C_{t_1}) \tag{9}$$

$$C_t = bCw \neq d \tag{10}$$

This can be plotted as a straight line with slope W/T on the equilibrium curve, Figure 2. It is called the operating line.
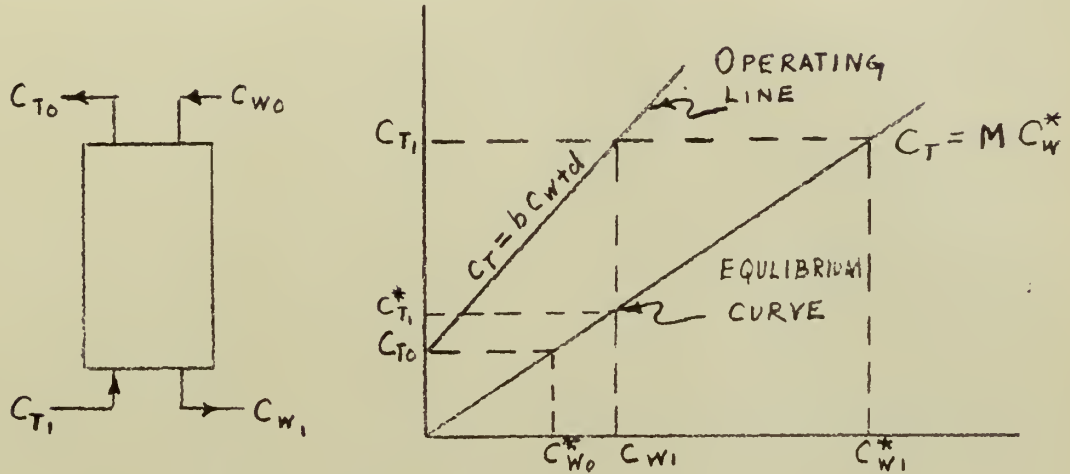
Figure 2. C_w equilibrium curve.

For a differential height in the column, dZ

$$N/\bullet = SWdC_w \tag{11}$$

substituting in (7)

$$SWdC_w = K_w a \ (C_w^* - C_w)SdZ \tag{12}$$

$$\frac{dC}{(C_w^* - C_w)} = \frac{K_w a dZ}{W} \tag{13}$$

and

$$\int_0^{C_{w_1}} \frac{dC_w}{(C_w^* - C_w)} = \frac{K_w a Z}{W} \tag{14}$$

The left hand side of the equation has been defined by Chilton and Colburn[8] as the number of transfer units, n, required to effect the extraction in question and the height of a transfer unit, HTU, is defined by

$$HTU_{ow} = \frac{Z}{\int_0^{C_{w_1}} \frac{dC_w}{(C_w^* - C_w)}} = \frac{Z}{\frac{K_w a Z}{W}} = \frac{W}{K_w a} \tag{15}$$

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Thus the HTU can be used to guage the effectiveness of a particular extraction. The greater the HTU, the higher the column which will be required, and vice versa.

The left hand side of equation (14) can be evaluated by graphical integration or it can be shown (appendix 1) that it is equal to $C_{w1}/(\Delta C_w)_{LM}$. Substituting in equation (14) and rearranging we see that

$$K_{wa} = WC_1/Z(\Delta C_w)_{LM} = N/\bullet SZ(\Delta C_w)_{LM} = N/\bullet V(\Delta C_w)_{LM} \quad (16)$$

Using similar reasoning, an equation for K_{ta} can be computed substituting t wherever the subscript w appears. The HTU can also be referred to the toluene side.

5. Scope of Thesis.

The present investigation was made on a pulsed packed liquid extraction column, extracting benzoic acid from toluene by means of distilled water. This system was selected because of the negligible mutual solubility of the liquid phases. In addition, at the concentrations utilized, the equilibrium curve is almost a straight line, permitting the use of analytical methods. Equilibrium data was available in articles by Feick and Anderson[5] and Appel and Elgin[9]. The data was checked for internal consistancy and a graph- Figure 5-was constructed using equilibrium data for 14.5°C, 19°C, and 24°C, taken from the latter reference and for 28.9°C, taken from the former.

Some of the variables which affect the effectiveness of extraction and therefore the height of a transfer unit, are density, viscosity, interfacial tension, which are system parameters. Column parameters which affect the HTU are toluene and water flow rates, packing size, pulse amplitude, pulse frequency, and the column diameter. Since only one system was studied, and in the temperature range being considered, the changes of density viscosity, and interfacial tension are relatively minor, the effects of these variables were not considered. The parameters which were varied were the pulse fre-

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quency and superficial water flow rate.

6. Experimental Equipment.

The column, which was built, is shown in Figure 3. It was subsequently modified by removing the air chamber on the top of the column and by feeding the extract to the top of the jackleg. These changes are reflected in the flow diagram, Figure 5. The column was a 3 foot high pyrex glass cylinder, with a 2 inch inner diameter, and was provided with flared outer edges to accommodate cast iron flanges. The lower settling chamber was a bell shaped glass section which was fifteen inches long with a 2 inch inner diameter at the top and a 4 inch inner diameter at the bottom. The upper settling chamber was a 12 inch long cylinder with a 4 inch inner diameter. It required a special stainless steel adaptor to permit its attachment to the packed section of the column.

The packing was 3/8 inch by 1/16 inch by 3/8 inch white porcelain Raschig rings, procured from the United States Stoneware Company of Akron, Ohio. The packing was dumped into the column which was partially filled with pulsing water. The packing settled during this pulsation and was restrained at the top by a 16 gage perforated stainless steel plate. The packing support plate was a 3/16 inch perforated stainless steel plate. The perforations in both plates were 1/8 inch in diameter.

The bellows was a 4 inch inner diameter and was made of teflon. It was manufactured by John L. Dore Inc. of Houston, Texas, and was mounted at the bottom of the column as shown in Figure 4. It was actuated by a cam which was cut to provide simple harmonic motion. It had a 6 inch diameter and a 1/4 inch amplitude which caused a pulse amplitude of 1-5/16 inches in the unpacked section of the column. The speed of the cam was geared down by a reduction ratio of 2:1. It was driven by a 3/4 horse power Vickers variable speed hydraulic transmission.
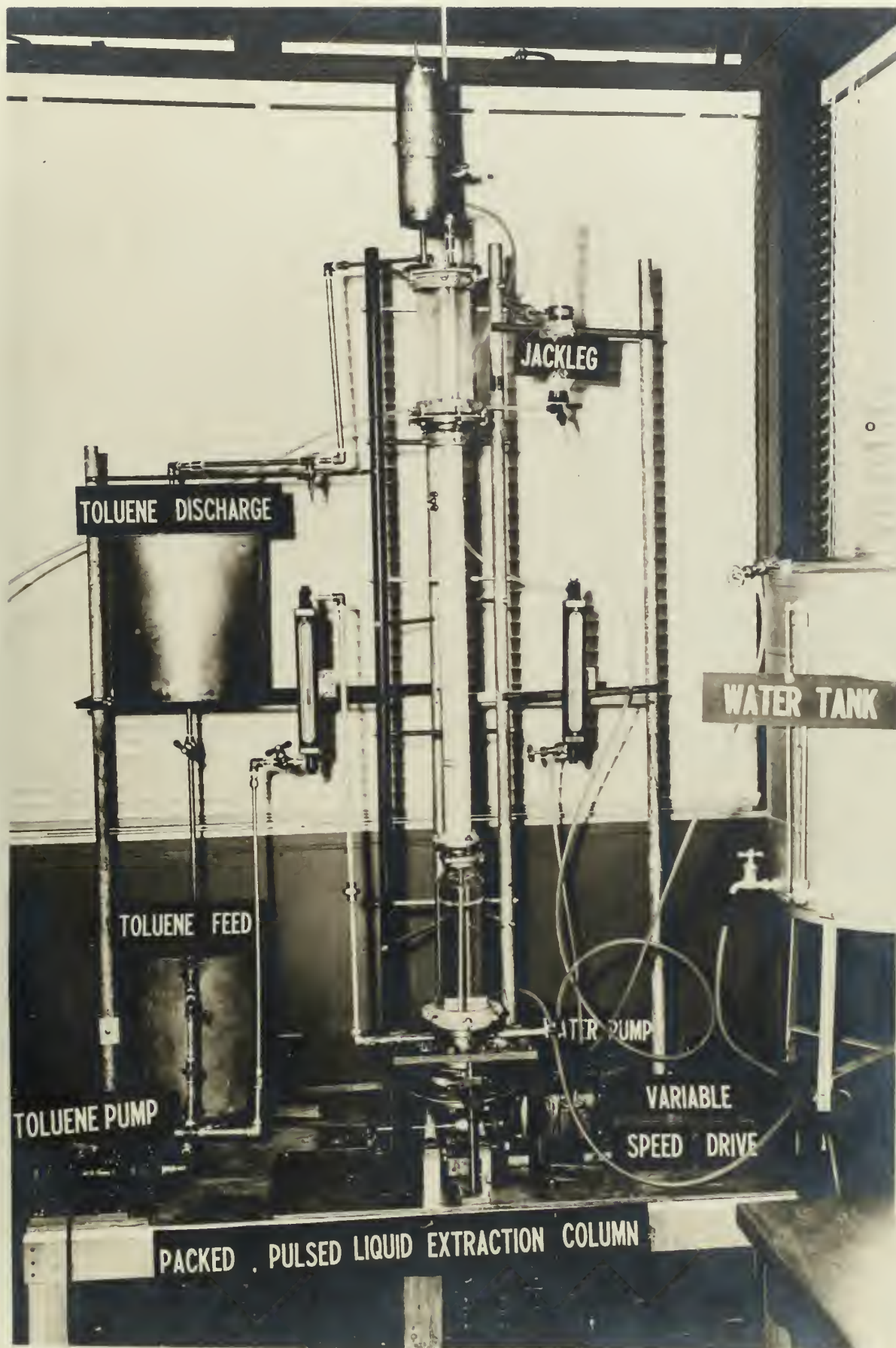
Figure 3 Page 14

The toluene tanks were 10 gallon capacity, and made of stainless steel. All metal which came into contact with the toluene phase was stainless steel. Both 1/4 inch i.p.s. pipe and 3/8 inch tubing were utilized on the toluene side. The toluene distributor was a shower type spray head, 1 inch in diameter with twelve holes drilled with a size 70 drill. The water side piping was 3/8 inch tygon tubing with nylon or hard rubber tube fittings.

The pumps were model D-11 stainless steel centrifugal pumps which were manufactured by Eastern Industries Inc. of Hamden, Connecticut. They were 1/4 horsepower and capable of delivering 5 gallons per minute at a pressure of 10 pounds per square inch. The rotameters were manufactured by Fisher and Porter and had a capacity of 0.148 gallons per minute, which corresponded to a superficial water flow rate of 54.4 cubic feet per hour per square foot of unpacked column area. Stainless steel check valves were placed between the rotometer and the column.

7. Design Considerations for Experimental Equipment, and Difficulties Experienced.

Stainless steel was used for all metal which came in contact with benzoic acid solutions because of their corrosive nature, which would result in ultimate contamination of the system which could result in the formation of stable emulsions. Since toluene attacks many organic packing and gasket materials, teflon was used throughout for this purpose. In this connection, the rubber packing rings at the ends of the rotameter tubes swelled so badly that they blocked the entire flow cross section of the rotameter. These were replaced by teflon which proved satisfactory.

A cam was used to actuate the bellows because if the amplitudes were changed by using different cams, it would be easy to reproduce the exact amplitude of one of the earlier cams if it were desired to do so. Consider-

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able difficulty was experienced in getting the cam to function properly. The friction between the cam and the follower caused the follower to bind on its guides. Splash lubrication was provided for the cam and a bronze oilite insert was placed in the follower at the point of contact. This helped somewhat but did not completely eliminate the problem. Making one of the follower guides more flexible, by cutting it free of its upper support and permitting it to act as a cantilever, finally solved the problem.

To reduce the load on the cam due to the compression of air in the discharge lines, an air chamber was placed at the top of the column. This resulted in the sucking back of air from the air chamber resulting in a disturbance of the interface. This problem was eliminated by extending the discharge line into the toluene settling chamber and permitting a small air pocket at the top of the column. The toluene syphoned out of the settling chamber only when the column was pulsed.

8. Experimental Procedure.

The toluene and the benzoic acid were both reagent grade and were procured from the General Chemical Division of the Allied Chemical and Dye Corporation. The water which was used for the solvent was distilled water saturated with toluene. The toluene feed was made up to about 0.0113 lb. mols of benzoic acid per cubic foot prior to beginning each run, and was saturated with water.

Before the column was assembled, the glass sections were cleaned with dichromate-acid cleaning solution and thoroughly rinsed with distilled water. This was to eliminate wetting of the glass by toluene and to help establish the water as the continuous phase. In addition, the packing was immersed in distilled water for the same reason. The column was then filled with water and when all of the packing was covered, toluene feed was introduced into the column. The cam was then operated to assist the toluene to

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Figure 4

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LEGEND

A. ROTOMETER
B. NEEDLE VALVE
C. GLOBE VALVE
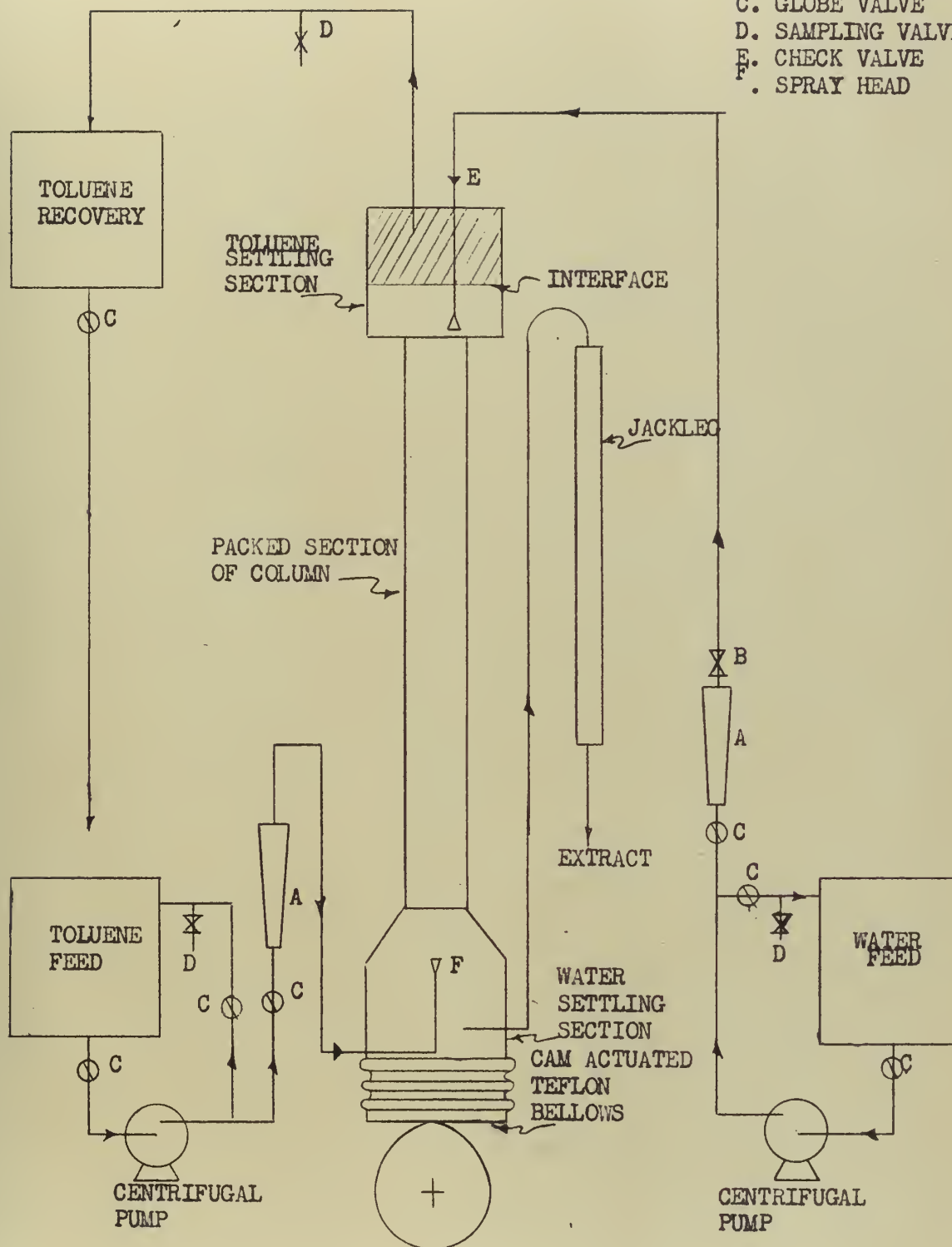D. SAMPLING VALVE
E. CHECK VALVE
F. SPRAY HEAD

FIGURE 5   FLOW DIAGRAM - LIQUID-LIQUID, PULSED, PACKED EXTRACTION COLUMN

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reach the top of the column to form a toluene layer in the upper settling chamber. When a sufficiently large layer of toluene was built up, the flow rates of the water and toluene were adjusted to the desired ones, the cam frequency was adjusted and the run was begun.

Since only the incoming flow rates were controlled by valves, it was necessary to maintain the level of the interface relatively constant in order to maintain a material balance. Adjusting the height of the jackleg permitted control of the interface level by controlling the rate of water phase discharge. The toluene phase automatically adjusted itself to suit the material balance. It was necessary to adjust the jackleg height by trial and error each time the flow rates or the frequency was changed, since at any particular height, the amount of water discharged into the jackleg decreased as the pulse frequency increased. If flooding occurred at the bottom of the column, control of the interface became virtually impossible. In this case the interface would rise and lowering the jackleg to lower the level of the interface would only aggrevate the situation since the flooding was due to too high a water discharge rate. The ultimate result was a building up of the toluene layer at the bottom and the disappearance of the toluene layer at the top.

As has been stated previously, flooding is a factor depending on the relative flow rates of the feed and solvent and represents one of the limiting factors on the capacity of the column. It is possible to induce premature flooding at flow rates far below the limiting rates. This may be due to improper adjustment of the jackleg or to improper design of the toluene spray head. The latter was found to be the case in the early runs. The distributor contained thirty 1/16 inch diameter holes. When the spray head mentioned in the section describing the equipment was used, this situation was materially improved. Other improvements were noticed when the spray

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head was placed as closely as possible to the packing support plate and by reducing the thickness of the support plate. Once the droplets of the dispersed phase entered the packed section of the column, they were formed and reformed by rushing up and down past the packing and their size was observed to be a function of the pulse frequency. The higher the frequency, the smaller the drops.

After the interface was maintained at a constant level, it was necessary to determine if steady state had been attained. This was determined by titrating the extract with a standard sodium hydroxide solution. After two succeeding samples, taken at five minute intervals after an initial thirty minutes of steady operation, proved to analyze within about 0.4% of each other, it was assumed steady state had been achieved. Final smaples of each stream were drawn off and each titrated three times. The average of the three determinations was the value used for omputation. The toluene samples were mixed with an equal volume of 95% ethanol, U.S.P. Navy Department Standard Stock number G51-A-1965. It was than titrated with an aqueous solution of sodium hydroxide using phenolphthalein indicator.

9. Sources of Error.

One of the sources of error is the assumption that steady state conditions have been achieved. There are several ways of checking to see if this has been accomplished. The first is to run check titrations on one of the exiting streams. The second is to run a material balance to see if the solute entering one phase is equal to the material leaving the other phase. In addition, cross plots of the data were made as in Figures 6 and 7. If the point did not fall on a smooth curve in both graphs, it was considered dubious and rechecked. Points not consistent with other data were discarded.

Another source of error was the fluctuation of flow rates due to the pulsing action of the column. It was attempted to have the rotameter float

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fluctuate about the desired value, particularly on the toluene side where the fluctuation was a little more pronounced. The water side rotameter was quite steady once the air was out of the system.

The temperature which was used to select the appropriate equilibrium curve was that of the toluene in the feed tank. A thermometer was immersed in the upper settling chamber and the most convenient place to check the lower end was to measure the temperature of the feed. There was a small temperature gradient across the column, but since several of the recheck runs were made at temperatures different from the original run, it is considered that this error was low.

Other minor sources of error are the calibration of the rotameters and the titrating errors both of which are well below the possible errors listed above.

10. Discussion of Results.

The results were computed in accordance with the procedure outlined in appendix 2 and recorded in appendices 3 and 4. The plan of experimentation which was followed, was to keep the toluene flow rate constant and low enough to permit a fairly wide variation of water flow rate without effecting flooding. It was possible to vary the water flow from 8.16 cu.ft./hr/ft to 32.65 cu.ft./hr/ft. The latter flow rate was at the point of incipient flooding and therefor was the limiting water flow rate when the toluene rate was 14.87 cu.ft./hr/ft. For each water flow rate the pulse frequency was varied from eleven to fifty cycles per minute. At fifty cycles per minute, a cloud of very fine droplets diffused through the water settling section of the column, and it was felt that this should be the maximum frequency at which the column should operate with the 1/4 inch amplitude cam.

The overall mass transfer coefficient and the height of a transfer unit was calculated with respect to the toluene phase and to the water phase,

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and are plotted in Figures 6 through 11. Since the toluene phase is the one which controls, it is considered that the values referred to this phase would give more significant results than those referred to the water phase. As can be seen, the curves based on the toluene side gave good families of curves with far less scatter of the data than those based on the water side. The curves and the data show, that the maximum flow rates and maximum pulse frequency resulted in the lowest values of HTU. There is reason to believe from the article by Chantry, Von Berg and Wiegandt,[6] that a minimum point on the HTU curve should be obtained prior to the flooding and that the HTU would increase till flooding occurred. Since their pulse amplitude was much smaller and thus the allowable frequencies much larger, it is possible that this effect could be detected using a smaller cam on this column.

The values of overall HTU ranged from .97-2.73 ft. when referred to water side and from 8.83-40.5 ft. when referred to the toluene side. For similar flow rates with an unpulsed column, Row, Koffolt, and Withrow [10] computed overall HTU referred to the water side ranging from 3.8 to 11.3 feet. Thus, it can be seen that pulsing a column increases the effectiveness of extraction about threefold. The only reference, which was found, in which a pulsed packed extraction column using benzoic acid, toluene and water was that of Feick and Anderson.[5] Their amplitudes were 3/16 inch in the unpacked section of the column and the pulse frequency was as high as 400 cycles per minute. At flow rates similar to the ones used in this thesis, their HTU, referred to the water side was 10 ft. for the unpulsed column and 0.66 ft. for an amplitude frequency produce of 56.3 inches per minute. This report lists an amplitude of 1.25 ft. for a pulse amplitude product of 52.5 inches per minute.

Figure 6

EFFECT ON HTU_{ot} ofVARIATION OF PULSE FREQUENCY-WITH CONSTANT WATER FLOWS AND CONSTANT TOLUENE FEED RATE OF 14.87 Cu.FT./Hr/FT^2.

$W = 8.16$ C.F.H./FT^2

$W = 16.32 \dfrac{CFH}{FT^2}$

$W = 24.42 \dfrac{C.F.H.}{FT^2}$

$W = 32.65 \dfrac{C.F.H.}{FT^2}$

HTU_{ot} - OVERALL HEIGHT OF TRANSFER UNIT REFERRED TO TOLUENE PHASE ∽ FT.

PULSE FREQUENCY ∽ CYCLES PER MINUTE

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EFFECT ON HTU_{ot} OF VARIATION OF WATER FLOW RATE, WITH CONSTANT TOLUENE FEED RATE OF 14.87 CU.FT./HR./FT^2 and CONSTANT PULSE FREQUENCIES

Figure 7   Page 23

HTU$_{ot}$ - OVERALL HEIGHT OF TRANSFER UNIT, REFERRED TO TOLUENE PHASE -FT.
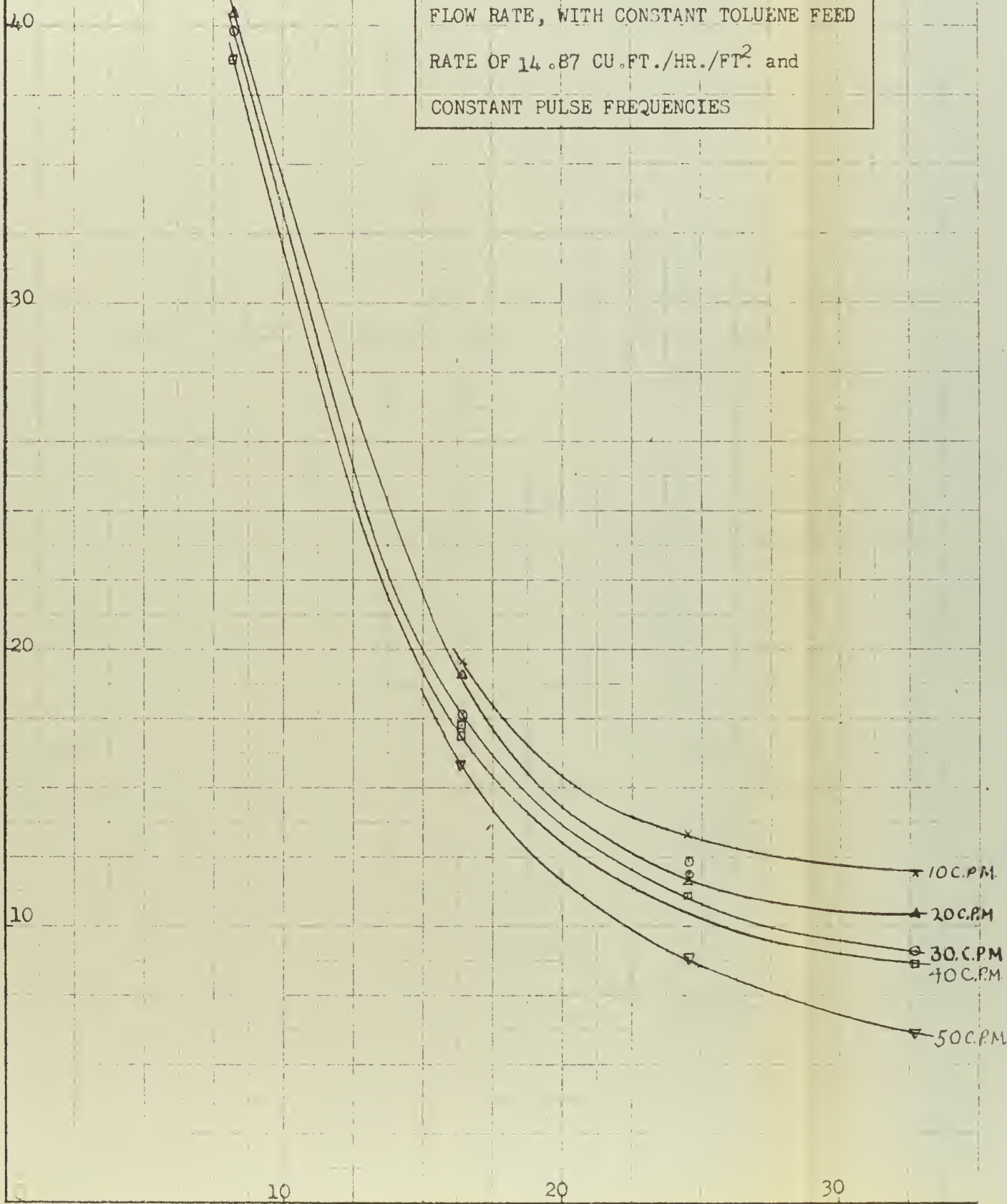
WATER FLOW RATE - CUBIC FEET/HOUR/SQUARE FOOT

10 C.P.M.
20 C.P.M
30. C.P.M
40 C.P.M.
50 C.P.M.

EFFECT ON $K_L a$ OF VARIATION OF PULSE
FREQUENCY, WITH CONSTANT FLOW OF
WATER AND CONSTANT TOLUENE FEED
RATE OF 14.87 Cu.Ft./Hr./Ft.^{2}

$W = 32.65$ Cu.FT./HR/FT.^{2}

$W = 24.48$ Cu.FT./HR/FT.^{2}

$W = 16.32$ Cu.FT./HR/FT^2

$W = 8.16$ Cu.FT/HR/FT^2

$K_t a$ - Lb. Mols/Hr./Cubic Ft./ Unit ΔC-OVERALL MASS TRANSFER COEFFICIENT

PULSE FREQUENCY -CYCLES PER MINUTE

Figure 8   Page 24

Figure 9

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EFFECT ON HTU_{ow} OF VARIATION OF PULSE FREQUENCY-WITH CONSTANT WATER FLOWS AND CONSTANT TOLUENE FEED RATE OF 14.87 CU.FT./HR./FT.^{2}

Figure 10

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EFFECT ON K_wa OF VARIATION OF PULSE
FREQUENCY, WITH CONSTANT FLOW OF
WATER AND CONSTANT TOLUENE RATE

K_wa — OVERALL MASS TRANSFER COEFFICIENT—Lb. Mols/Hr./Cubic Ft./Unit ΔC

PULSE FREQUENCY — CYCLES PER MINUTE

W = 32.65

N = 24.48

W = 16.32

W = 8.16
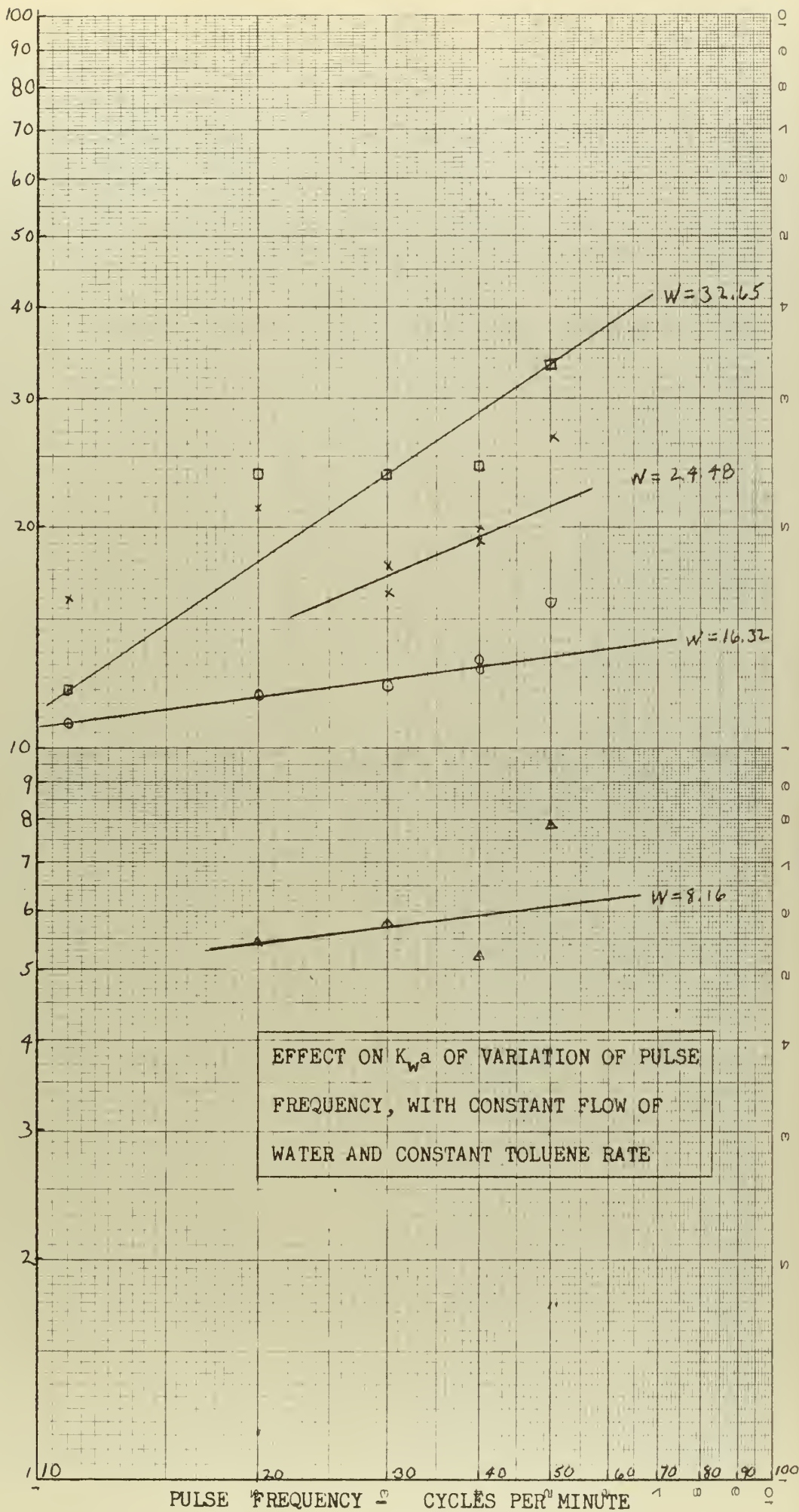
Figure 11   Page 27

11. Conclusions.

a. It may therefore be concluded that pulsing will greatly improve the effectiveness of extraction.

b. Within the range of flow rates, pulse frequencies and amplitudes investigated, the higher the water flow rate and the greater the frequency of pulsing, the lower the HTU, and the greater the amount of mass transfer. The limiting factors were flooding and formation of emulsions at high pulse frequencies.

12. Future Work.

Very little has been published on the effect of using various size packing on the performance of a pulsed packed column. Four sizes of packing was procured for use in this column but time did not permit investigation along these lines. In addition, the effect of changing pulse amplitude should be investigated. Lower amplitudes would permit the use of a wider range of pulse frequencies which might bring out an optimum condition for pulsing.

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APPENDIX **1** - Derivation of Expression Involving Logarithmic Mean Driving Force.

Reference is made to equations (1) through (16), which appear on pages 8 through 12 in the text.

Equating the values for C_t in equations (6) and (10) we get

$$M C_w^* = b C_w + d \tag{17}$$

or

$$C_w^* = \frac{b}{M} C_w + \frac{d}{M} \tag{18}$$

When $\quad C_w = 0, \quad C_{wo} = \frac{d}{M} \tag{19}$

$$\int_0^{C_{w_1}} \frac{d C_w}{C_w^* - C_w} = \int_0^{C_{w_1}} \frac{d C_w}{b/_M C_w + d/_M - C_w} = \int_0^{C_{w_1}} \frac{M \, d C_w}{(b - M) C_w + d} \tag{20}$$

$$= \frac{M}{b-M} \ln \frac{C_{w_1}(b-M) + d}{d} = \frac{M}{b-M} \ln \frac{b/_m C_{w_1} + d/_m - C_{w_1}}{d/m} \tag{21}$$

$$= \frac{M}{b-M} \ln \frac{C_{w_1}^* - C_{w_1}}{C_{wo}} = \frac{M}{b-M} \ln \frac{\Delta C_{w_1}}{C_{wo}} \tag{22}$$

$$= \frac{M}{b-M} \frac{\Delta C_{w_1} - C_{wo}}{\Delta C_{w_1} - C_{wo}} \ln \frac{\Delta C_{w_1}}{C_{wo}} \tag{23}$$

If $(\Delta C_w)_{LM}$ IS defined by

$$(\Delta C_w)_{LM} = \frac{\Delta C_{w_1} - C_{wo}}{\ln \left(\frac{\Delta C_{w_1}}{C_{wo}}\right)} \tag{24}$$

Then $\int_0^{C_{w_1}} \frac{d C_w}{C_w^* - C_w} = \frac{M}{b-M} \frac{\Delta C_{w_1} - C_{wo}}{(\Delta C_w)_{LM}} \tag{25}$

$$= \frac{M(b/_M C_{w_1} + d/_M) - M C_{w_1} - \frac{Md}{M}}{(b-M) (\Delta C_w)_{LM}} \tag{26}$$

$$= \frac{(b-M) C_{w_1}}{(b-M)(\Delta C_w)_{LM}} = \frac{C_{w_1}}{(\Delta C)_{LM}} \tag{27}$$

EQUILIBRIUM DISTRIBUTION OF BENZOIC
ACID BETWEEN TOLUENE AND WATER.

14.5 °C, 19°C, 24°C Curves – ref. 9
28.9 C curve – ref. 5

CONCENTRATION OF BENZOIC ACID IN TOLUENE, LB. MOLS./ CUBIC FOOT. (CT)

CONCENTRATION OF BENZOIC ACID IN WATER, LB. MOLS./ CUBIC FOOT. (C_W)

Figure 12 Page 31
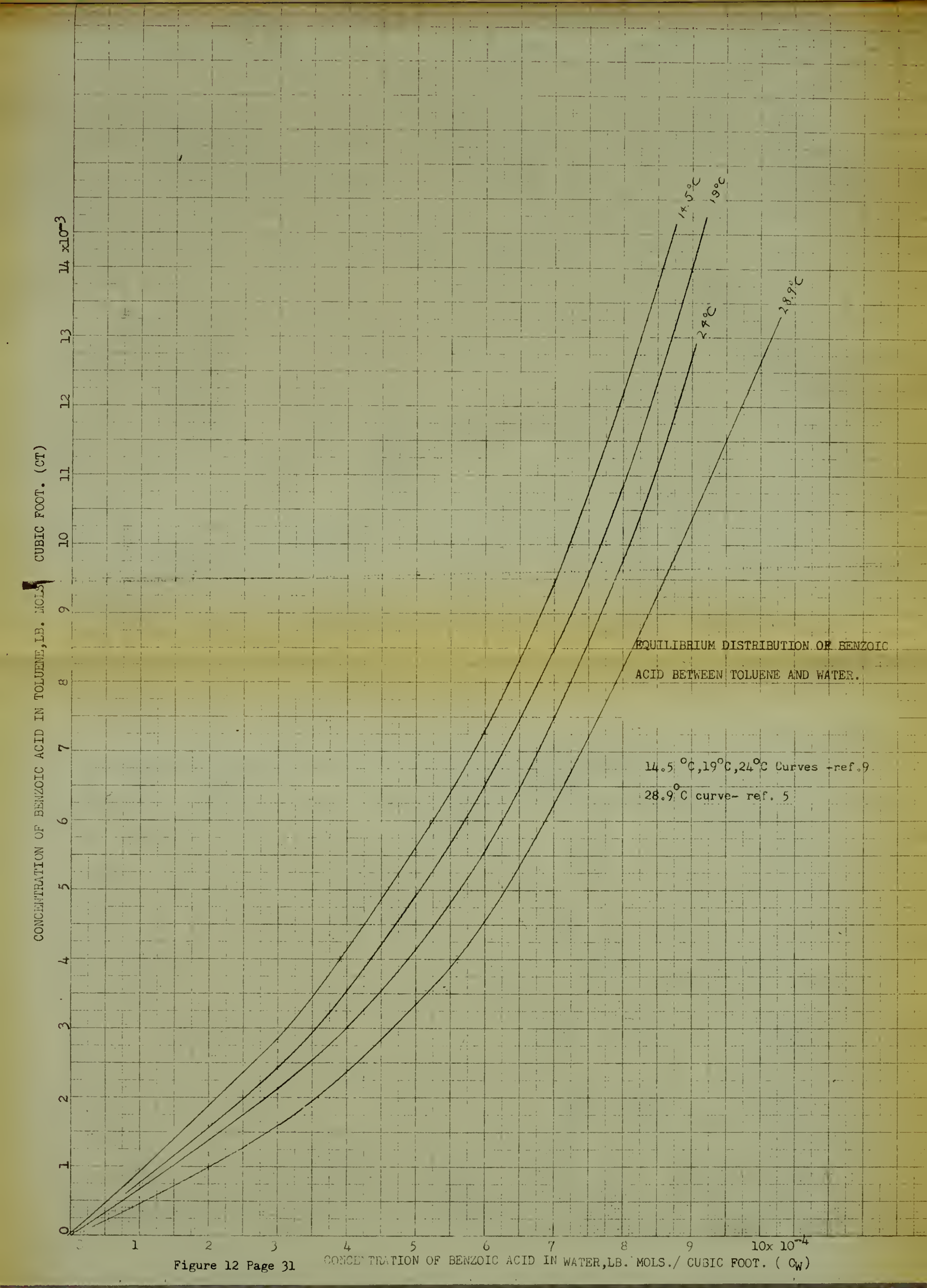
Column Dia. was 2 in. ; S = 0.02182 sq. Ft.   Z = 3Ft.   V = 0.06546 Ft^3

The results of run #14 will be calculated. The temperature of the

toluene feed was 24.2°C and the 24° curve from figure 12 was used to

select the equilibrium data.

1. Material Balance:

  Benzoic acid removed from toluene side

$$\frac{N}{\theta S} = T (C_{T_1} - C_{T_0} = 14.87 \frac{ft^3}{hr-ft^2} \times (11.36 - 10.04) \times 10^{-3} \frac{\#mol}{ft^3}$$

$$= 14.87 \times 1.32 \times 10^{-3} = 19.76 \times 10^{-3} \frac{\#\, mol}{hr-ft^2}$$

$$\frac{N}{\theta S} = W (C_{W_1} - C_{W_0}) = 24.48 (8.22 - 0) \times 10^{-4}$$

$$= 20.34 \times 10^{-3} \frac{\#\, mol}{hr\, ft^3} \qquad \left(\frac{N}{\theta S}\right)_{mean} = 19.95 \times 10^{-3} \frac{\#\, mol}{ft^3}$$

THESE   VALUES   WERE WITHIN 1.82% OF each other

By equation #16

$$K_{Wa} = \frac{N}{\theta V} \times \frac{1}{(\Delta C_W)_{LM}} \quad ; K_{Ta} = \frac{N}{\theta V} \times \frac{1}{(\Delta C_T)_{LM}}$$

$$(N/\theta V) = \left(\frac{N}{\theta S Z}\right) = \frac{19.95 \times 10^{-3}}{3} = 6.65 \times 10^{-3} \frac{\#\, mol}{ft^3}$$

END	$C_T \times 10^3$	$C_W \times 10^4$	$C_W^* \times 10^4$	$(C_W^* - C_W) \times 10^4$
0	10.04	0	8.10	8.10
1	11.36	8.22	8.59	.37

$$(\Delta C_W)_{LM} = \frac{8.10 - .37 \times 10^{-4}}{\ln \frac{8.10 \times 10^{-4}}{.37 \times 10^{-4}}} = \frac{7.73 \times 10^{-4}}{\ln 21.89}$$

$$= \frac{7.73 \times 10^{-4}}{3.09} = 2.50 \times 10^{-4} \frac{\#\, mole}{ft^3}$$

$$K_{Wa} = \frac{6.65 \times 10^{-3}}{2.5 \times 10^{-4}} = 26.6 \frac{\#\, mole}{hr - ft^3\, UNIT\, \Delta C}$$

$$HTU_{OW} = \frac{W}{K_{Wa}} = \frac{24.48}{26.6} = 0.921\, ft$$

FOR TOLVENE SIDE

END	$C_T \times 10^3$	$C_W \times 10^4$	$C_t^* \times 10^3$	$(C_T - C_T^*) \times 10^3$
0	10.09	0	0	10.09
1	11.36	8.22	10.33	1.03

$$\Delta = 9.01 \times 10^{-3}$$

$$(\Delta C_T)_{LM} = \frac{(10.09 - 1.03) \times 10^{-3}}{\ln \frac{10.09 \times 10^{-3}}{1.03 \times 10^{-3}}} = \frac{9.01 \times 10^{-3}}{\ln 9.75}$$

$$= \frac{9.01 \times 10^{-3}}{2.277} = 3.95 \times 10^{-3} \frac{\# \, mol}{ft^3}$$

$$K_{T9} = \frac{N}{\theta v} \times \frac{1}{(\Delta C_T)_{LM}} = \frac{6.65 \times 10^{-3}}{3.95 \times 10^{-3}}$$

$$= 1.683 \frac{\# \, mol}{hr - ft^3 \, unit \, \Delta C}$$

$$HTU_{OT} = \frac{I}{K_{T9}} = \frac{14.87}{1.683} = \underline{\underline{8.83 \, ft}}$$

APPENDIX 3 – Raw data.

Run No.	Temp. °C	Cycles per Minute	T	W	C_{t1} x10³	C_{to} x10³	C_{wl} x10⁴	$\frac{N}{\theta V}$ x10³	$(C_w)_{LM}$ x10⁴	$(C_t)_{LM}$ x10³	K_{wa}	K_{ta}	HTU_{ow}	HTU_{ot}
1 a		15	11.95	13.60	7.86	6.93	7.34	3.52						
2 a		17.5	11.95	13.60	8.93	8.15	7.13	3.06						
3	19	24	11.95	13.60	9.50	8.50	7.61	3.71	2.19	2.91	16.95	1.28	.80	9.37
4 b	22.0	50	11.95	13.60	11.38	10.33	8.18	3.95	2.63	4.29	15.01	.82	.91	14.66
5 b	19.9	25	14.87	19.04	11.54	10.44	8.12	5.30	2.80	4.58	18.94	1.16	1.01	12.89
6	19.5	50	14.87	16.32	11.34	10.43	8.09	4.45	2.81	4.57	15.82	.97	1.03	15.28
7	27.0	50	14.87	16.32	11.43	10.50	8.19	4.53	3.47	5.37	13.07	.84	1.25	17.62
8	30.5	20	23.87	22.83	11.45	10.68	8.12	5.99	4.08	5.82	14.69	1.01	1.56	23.56
9	25.3	37	23.87	24.48	11.30	10.48	8.06	6.61	2.69	4.46	24.56	1.48	1.00	16.12
10	23.0	52	23.87	24.48	11.41	10.71	7.88	5.55	2.82	4.80	19.66	1.16	1.25	20.67
11	25.0	20	14.87	24.48	11.24	9.93	8.13	6.56	2.57	4.07	25.54	1.61	.96	9.22
12	23.1	31	14.87	24.48	10.94	9.70	8.08	6.37	2.44	3.80	26.11	1.68	.94	8.87
13	30.0	40	14.87	24.48	10.99	9.67	8.31	6.62	3.52	4.98	19.00	1.34	1.29	11.11

a. Temp. not observed – runs not calculated
b. Phase reversal occurred during run

APPENDIX 3 - Raw data.
Continued

Run No.	Temp. °C	Cycles per Minute	T	W	C_{t1} x10³	C_{to} x10³	C_{w1} x10⁴	$\frac{N}{\sigma V}$ x10³	$(C_w)_{LM}$ x10⁴	$(C_t)_{LM}$ x10³	K_{wa}	K_{ta}	HTU_{ow}	HTU_{ot}
14	24.2	50	14.87	24.48	11.36	10.04	8.22	6.65	2.50	3.95	26.59	1.68	.92	8.83
15	33.5	30	14.87	24.48	11.39	9.96	8.53	7.02	4.31	5.84	16.30	1.20	1.50	12.36
16	29.2	11	14.87	24.48	11.29	9.98	7.99	6.51	4.07	5.86	15.99	1.11	1.53	13.38
17	26.7	11	14.87	32.65	10.47	9.28	5.92	6.17	5.14	7.28	11.97	.85	2.73	17.60
18	25.5	20	14.87	32.65	11.37	9.84	7.52	7.88	3.47	5.56	23.72	1.42	1.38	10.49
19	30	30	14.87	32.65	11.41	9.69	8.22	8.74	3.68	5.42	23.78	1.61	1.37	9.22
20 c	28.9	40	14.87	32.65	11.31	9.55	8.32	8.89	3.66	5.19	24.29	1.71	1.34	8.68
21	25.8	50	14.87	32.65	11.31	9.68	8.16	8.48	2.53	3.53	33.53	2.41	.97	6.18
22	28.0	30	14.87	14.32	11.31	10.38	8.33	4.57	3.78	5.45	12.11	.84	1.34	17.72
23	25.4	20	14.87	16.32	11.29	10.28	8.05	4.69	2.74	4.45	17.16	1.06	.95	14.09
24	26.6	20	14.87	16.32	11.34	10.49	7.97	4.28	3.63	5.51	11.79	.78	1.38	19.15
25	26.1	40	14.87	16.32	11.35	10.49	8.17	4.36	3.40	4.96	12.81	.88	1.27	16.91
26	30	11	14.87	16.32	11.35	10.46	8.17	4.43	4.11	5.85	10.77	.76	1.52	19.64

c. Point of incipient flooding

APPENDIX 3 – Raw data.
Continued

Run No.	Temp. °C	Cycles per Minute	T	W	C_{t1} x10³	C_{to} x10³	C_{wl} x10⁴	$\frac{N}{\theta V}$ x10³	$(C_w)_{LM}$ x10⁴	$(C_t)_{LM}$ x10³	K_{wa}	K_{ta}	HTU_{ow}	HTU_{ot}
27	30	30	14.87	8.16	11.30	10.86	8.14	2.21	3.83	5.95	5.77	.37	1.41	39.90
28	25	50	14.87	8.16	11.29	10.90	8.16	2.18	2.78	5.97	7.85	.36	1.04	40.70
29	29	20	14.87	8.16	11.29	10.94	8.16	2.21	4.06	5.99	5.45	.37	1.50	40.50
30	25	40	14.87	8.16	11.31	10.99	8.03	2.18	4.17	5.71	5.24	.38	1.56	38.80
31	26.7	40	14.87	24.48	11.31	10.06	8.27	6.40	3.22	4.57	19.89	1.34	1.23	11.09
32	29.2	30	14.87	24.48	11.32	10.05	8.36	6.54	3.68	5.26	17.75	1.24	1.38	11.96
33	24.6	20	14.87	24.48	11.20	10.06	7.87	6.33	3.06	4.84	20.65	1.31	1.19	11.37
34	26.3	40	14.87	16.32	11.44	10.38	8.17	4.25	3.22	4.97	13.32	.86	1.23	17.34

APPENDIX 4- Reduced Data

Run No.	Cycles per minute	T	W	$K_t a$	HTU_{ot}	$K_w a$	HTU_{ow}
29	20	14.87	8.16	.370	40.50	5.45	1.50
27	30	14.87	8.16	.372	39.90	5.77	1.41
30	40	14.87	8.16	.382	38.80	5.24	1.56
28	50	14.87	8.16	.364	40.70	7.85	1.04
26	11	14.87	16.32	.757	19.64	10.77	1.515
24	20	14.87	16.32	.776	19.15	11.79	1.384
22	30	14.87	16.32	.839	17.72	12.11	1.348
25	40	14.87	16.32	.879	16.91	12.81	1.274
34	40	14.87	16.32	.857	17.34	13.32	1.225
6	50	14.87	16.32	.973	15.38	15.82	1.249
16	11	14.87	24.48	1.111	13.38	15.99	1.531
33	20	14.87	24.48	1.308	11.37	20.65	1.185
15	30	14.87	24.48	1.203	12.36	16.30	1.502
32	30	14.87	24.48	1.242	11.94	17.75	1.381
13	40	14.87	24.48	1.338	11.11	19.00	1.288
31	40	14.87	24.48	1.341	11.09	19.89	1.251
14	50	14.87	24.48	1.683	8.83	26.59	.921
17	11	14.87	32.65	.845	17.60	11.97	2.728
18	20	14.87	32.65	1.418	10.49	23.72	1.376
19	30	14.87	32.65	1.612	9.22	23.78	1.373
20	40	14.87	32.65	1.713	8.68	24.29	1.344
21	50	14.87	32.65	2.406	6.18	33.53	.974

APPENDIX 5 – Nomenclature

Symbol	Definition

a' Total interfacial area, sq. ft.

a Interfacial area/unit volume of column, sq. ft./ cu. ft.

b Slope of operating line, W/T.

C Concentration of benzoic acid in either water or toluene phase, lb. mols/ cu. ft.

D Molecular diffusivity or diffusion Coefficient, sq. ft./hr.

d Intercept of operating line, $(W/T)C_{w1}-C_{t1}$

E Eddy diffusivity, $ft^2/hr.$

HTU   Height of one transfer unit, ft.

K_a Overall mass transfer coefficient, lb mols/hr/cu.ft./unit C.

k Mass transfer coefficient, lb mols/hr/cu.ft./unit C.

L Distance in direction of diffusion, ft.

M Distribution coefficient, slope of equilibrium curve C_t/C_w^*.

$N\,/\theta$ Lb Mols of benzoic acid transferred per hour.

S Cross-sectional area of unpacked column, sq. ft.

T Superficial velocity of toluene phase, cu. ft./ hr/S , cu.ft./hr/ft^2.

V Volume of column, cu.ft.

W Superficial velocity of water phase, cu.ft./hr/S, cu. ft./hr/ft.^{2}

Z Height of column, ft.

     Difference between value and equilibrium value.

C $C-C^*$ OR $C^* -C$

$(C)_{LM}$ Logarithmic mean driving force, lb mols/cu. ft.

Subscripts

i interface

Symbol   Definition

Subscripts:

o   Top of column, or overall.

l   Bottom of column.

t   Toluene phase.

w   Water phase.

Superscript

*   Equilibrium value.